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WO 01/15537 A1

(54) Title: *MORINDA CITRIFOLIA OIL*

(57) **Abstract:** An essential oil product obtained from the Indian mulberry *Morinda citrifolia* plant and the process of extracting and purifying the oil is disclosed. According to one embodiment, the seeds from the Indian mulberry fruit are dried, preferably to a moisture content less than 10%. The seeds are ground or shredded to facilitate the removal of natural occurring oil. The shredded or ground seed flakes are pressed to expel *Morinda citrifolia* oil. The remaining seed cake is then mixed with a food grade, non-polar extraction solvent such as hexane. The mixture is heated for a sufficient length of time to complete the extraction process. The extraction solvent is then evaporated from the mixture leaving the *Morinda citrifolia* oil. The oil is further refined, bleached, dried, and deodorized to remove free fatty acids and other unwanted components. An antioxidant can optionally be added to stabilize the oil for further processing or packaging.

***MORINDA CITRIFOLIA OIL***Field of the Invention

5 The present invention relates an essential oil product obtained from the *Morinda citrifolia* plant and to the process of extracting and purifying the oil.

Background of Invention

10 The Indian Mulberry plant, known scientifically as *Morinda citrifolia L.*, is a shrub, or small or medium sized tree 3 to 10 meters high. It grows in tropical coastal regions around the world. The plant grows randomly in the wild, and it has been cultivated in 15 plantations and small individual growing plots. The Indian mulberry plant has somewhat rounded branches and evergreen, opposite (or spuriously alternate), dark, glossy, wavy, prominently-veined leaves. The leaves are broadly elliptic to oblong, pointed at both ends, 10-30 cm in length and 5-15 cm wide.

15 The Indian mulberry flowers are small, white, 3 to 5 lobed, tubular, fragrant, and about 1.25 cm long. The flowers develop into compound fruits composed of many small drupes fused into an ovoid, ellipsoid or roundish, lumpy body, 5-10 cm long, 5-7 cm thick, with waxy, white or greenish-white or yellowish, semi-translucent skin. The fruit contains 20 "eyes" on its surface, similar to a potato. The fruit is juicy, bitter, dull-yellow or yellowish-white, and contains numerous red-brown, hard, oblong-triangular, winged, 2-celled stones, each containing 4 seeds.

25 When fully ripe, the fruit has a pronounced odor like rancid cheese. Although the fruit has been eaten by several nationalities as food, the most common use of the Indian mulberry plant was as a red and yellow dye source. Recently, there has been an interest in the nutritional and health benefits of the Indian mulberry plant.

It would be a significant advancement in the art to provide the essential oil from the Indian mulberry plant and to provide a process for obtaining the essential oil from the Indian mulberry plant.

30

Summary of the Invention

The present invention is directed to an essential oil extracted from Indian mulberry (*Morinda citrifolia*) seeds and to a process of extracting the oil. The Indian mulberry oil typically includes a mixture of several different fatty acids as triglycerides, such as

palmitic, stearic, oleic, and linoleic fatty acids, and other fatty acids present in lesser quantities.

The essential oil preferably includes an antioxidant to inhibit spoilage of the oil. Conventional, food grade antioxidants are preferably used, including, but not limited to, 5 tocopherol acetate (vitamin E acetate), propyl gallate, TBHQ (t-butyl hydroquinone), BHT (3,5-bis(t-butyl)-4-hydroxytoluene), and French maritime pine bark extract.

The process for extracting Indian mulberry oil from its seeds typically involves 10 obtaining Indian mulberry seeds that are dried to a moisture content less than 10%, by weight, and preferably from 5% to 8%, by weight. The seeds are cracked, ground, flaked, shredded, or mechanically reduced in size, typically from 2 to 20 mm. As used herein, 15 ground seeds include seeds that may be ground, cracked, flaked, shredded, or otherwise reduced in size. The ground seeds are then pressed in a mechanical press or screw press to expel some of the oil. The remaining seed cake is mixed with a nonpolar extraction solvent. The extraction solvent is preferable food-grade. One currently preferred nonpolar solvent is hexane.

Oil extraction with the nonpolar solvent preferably takes place at a temperature range between 80°F and 200°F and ranges from a time of 30 minutes up to 12 hours. It is presently preferred to complete the extraction step as quickly as possible. The nonpolar solvent is preferably removed from the extracted oil through flash evaporation.

20 The oil from the mechanical press and from the nonpolar solvent extraction may be combined or further refined separately. The refining process removes impurities from the oil. Caustic, water, and phosphoric acid can be added to the oil to precipitate impurities. The precipitating agents and oil are mixed and heated to a temperature between 100°F and 200°F, the currently preferred target temperature is between 170°F and 180°F. The mixture 25 is centrifuged to remove precipitated impurities that include, but are not limited to, insoluble gums, phosphatides, and free fatty acids.

The oil can optionally be further refined by bleaching and deodorizing. Bleaching involves the addition of one or more filtering agents to the oil. The oil is filtered through a mechanical filter to remove the filtering agents. Currently preferred filtering agents 30 include diatomaceous earth (D.E.) and activated charcoal. Bleaching removes color bodies and soap materials generated during the refining process. Bleaching is preferably done at temperatures between 100°F and 200°F.

The oil may or may not be vacuumed dried to remove any residual water. The oil is deodorized to remove volatile impurities. This involves steam distillation under a vacuum and at temperatures between 300°F and 500°F. An antioxidant can be included with the extracted oil, as mentioned above.

5

#### Detailed Description of the Invention

The present invention is directed to an essential oil product obtained from the Indian mulberry (*Morinda citrifolia*) plant and to the process of extracting and purifying the oil. According to one currently preferred embodiment within the scope of the present invention, the oil is extracted from Indian mulberry seeds. The seeds from the Indian mulberry fruit are dried and then packed in large containers for storage and transport. The seeds can be processed immediately or they can be stored for a period of time, usually up to about one year.

The seeds are preferably dried because residual water can contaminate the extraction solvent, which is oil based. Also the presence of water in the extraction process can cause oxidation of the oil. The rate of drying is preferably quick enough to be economical and slow enough to protect the oil from heat degradation. Drying usually takes from 10 to 100 days. The seeds are preferably dried in the sun or by a mechanical drier. The seeds are preferably dried to a moisture content less than 10%, by weight, and more preferably from about 5% to 8%, by weight. Conventional moisture detectors can be used to measure the moisture content of the seeds during and after the drying process.

The seeds are cracked, ground, flaked, shredded, or mechanically reduced in size in order to facilitate the removal of natural occurring oil. As mentioned above, the term ground seeds include seeds that are ground, cracked, flaked, shredded, or otherwise reduced in size. The seeds are preferably ground through a grinder or flaker with a grinder plate size between about 2 mm to 20 mm hole size. The seeds are optimally reduced to a size falling in this range. The ground seed size is important because it effects the yield and quality of the extracted oil. Too large a flake size decreases the yield. Too small a flake size leaves a lot of undesired seed components in the oil.

The ground seeds are preferably pressed in a mechanical press or screw press to expel some of the oil. The remaining seed cake is mixed with a nonpolar, food grade extraction solvent. Hexane is a currently preferred nonpolar solvent. The mixture of nonpolar solvent and seed cake is preferably heated to a temperature between 80°F and

200°F and for a time from about 30 minutes up to 12 hours. The nonpolar solvent mixes with the Indian mulberry oil remaining in the seeds and carries the oil out of the water-based seed material matrix.

5 Indian mulberry oil pressed from the seeds and from the hexane extraction may be combined or further refined separately to remove impurities from the oil. Such unwanted impurities include, but are not limited to, insoluble gums, phosphatides, and free fatty acids. Caustic, water, and phosphoric acid are currently preferred precipitating agents that can be added to the oil to precipitate impurities. The oil and precipitating agent mixture is preferably heated to a temperature between 100°F and 200°F, and more preferably heated 10 to a temperature between 170°F to 180°F. The mixture is preferably centrifuged to remove the precipitated impurities.

15 The oil may be optionally further refined by bleaching and deodorizing. Bleaching involves the addition of one or more filtering agents to the oil to remove color bodies and soap materials generated during the refining process. Currently preferred filtering agents include, but are not limited to diatomaceous earth (D.E.) and activated charcoal. The oil is 20 filtered through a mechanical filter to remove the filtering agents. Bleaching is done at temperatures between 100°F and 200°F. Residual water can be removed from the oil by vacuum drying.

25 The oil is preferably deodorized to remove volatile impurities. This involves steam distillation under a vacuum. Typical distillation temperatures range from 300°F and 500°F.

An antioxidant can optionally be added to stabilize the oil for further processing or 20 packaging. Suitable antioxidants include, but are not limited to, tocopherol acetate (vitamin E acetate), propyl gallate, TBHQ (t-butyl hydroquinone), BHT (3,5-bis(t-butyl)-4-hydroxytoluene), French maritime pine bark extract. The *Morinda citrifolia* seed oil may 25 be used in, but is not limited to massage oil, cosmetics, candles, and as a product by itself.

The fragrance of the oil ranges from a nutty, mellow fragrance to a slightly sharp chemical solvent fragrance. The extracted oil appearance is from a rich golden yellow to a brownish tan color with a clear transparency, preferably free of foreign material or particulates.

30 From an analysis of the *Morinda citrifolia* oil, the oil is mainly composed of fatty acids as triglycerides. The free fatty acid content typically ranges from 5 to 9 grams/100 grams oil. The peroxide value typically ranges from 3 to 6 MEQ/KG, and the vitamin E

content typically ranges from 11 to 25 IU/100 gram. The approximate fatty acid composition of the oil is reported below in Table 1.

Table 1

	<u>Fatty Acid</u>	<u>Wt. %</u>
5	Caprylic	0.5
	Palmitic	8.4
	Stearic	4.0
	Oleic	13.8
	Linoleic	66.8
10	Linolenic	0.2
	Arachidic	0.5
	Eicosenoic	0.2
	Other fatty acids	5.6

15 The foregoing concentrations in the extracted oil can vary from one batch to another, but the variations generally do not exceed  $\pm 10\%$ , by weight.

## CLAIMS:

1. An essential oil comprising oil extracted from *Morinda citrifolia* seeds.
2. An essential oil according to claim 1, further comprising an antioxidant.
- 5 3. An essential oil according to claim 2, wherein the antioxidant is selected from tocopherol acetate (vitamin E acetate), propyl gallate, TBHQ (t-butyl hydroquinone), BHT (3,5-bis(t-butyl)-4-hydroxytoluene), and French maritime pine bark extract.
- 10 4. An essential oil according to claim 1, comprising palmitic, stearic, oleic, and linoleic fatty acids.
5. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds is bleached.
- 15 6. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds is deodorized.
7. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds comprises a mixture of fatty acids selected from caprylic, palmitic, stearic, oleic, linoleic, linolenic, arachidic, and eicosenoic fatty acid.
- 20 8. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds has a free fatty acid content ranging from 5 to 9 grams/100 grams oil.
- 25 9. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds has a peroxide value ranging from 3 to 6 MEQ/KG.
- 30 10. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds has a vitamin E content ranging from 11 to 25 IU/100 gram.

11. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds is an ingredient in massage oil.

5 12. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds is an ingredient in cosmetics.

13. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds is an ingredient in candles.

10 14. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds comprises a mixture of caprylic, palmitic, stearic, oleic, linoleic, linolenic, arachidic, and eicosenoic fatty acid.

15 15. An essential oil according to claim 1, wherein the oil extracted from *Morinda citrifolia* seeds comprises:

0.5 wt. % caprylic fatty acid;  
8.4 wt. % palmitic fatty acid;  
4.0 wt. % stearic fatty acid;  
13.8 wt. % oleic fatty acid;  
66.8 wt. % linoleic fatty acid;  
0.2 wt. % linolenic fatty acid;  
0.5 wt. % arachidic fatty acid; and  
0.2 wt. % eicosenoic fatty acid;

wherein the foregoing concentrations can vary up to  $\pm 10\%$ , by weight.

25 16. An essential oil comprising:

oil extracted from *Morinda citrifolia* seeds containing a mixture of fatty acids selected from caprylic, palmitic, stearic, oleic, linoleic, linolenic, arachidic, and eicosenoic fatty acid, wherein said extracted oil has a free fatty acid content ranging from 5 to 9 grams/100 grams oil; and  
an antioxidant.

17. An essential oil according to claim 16, wherein the antioxidant is selected from tocopherol acetate (vitamin E acetate), propyl gallate, TBHQ (t-butyl hydroquinone), BHT (3,5-bis(t-butyl)-4-hydroxytoluene), and French maritime pine bark extract.

5 18. An essential oil according to claim 16, wherein the oil extracted from *Morinda citrifolia* seeds is bleached.

19. An essential oil according to claim 16, wherein the oil extracted from *Morinda citrifolia* seeds is deodorized.

10 20. An essential oil according to claim 16, wherein the oil extracted from *Morinda citrifolia* seeds has a peroxide value ranging from 3 to 6 MEQ/KG.

15 21. An essential oil according to claim 16, wherein the oil extracted from *Morinda citrifolia* seeds has a vitamin E content ranging from 11 to 25 IU/100 gram.

22. An essential oil according to claim 16, wherein the oil extracted from *Morinda citrifolia* seeds is an ingredient in massage oil.

20 23. An essential oil according to claim 16, wherein the oil extracted from *Morinda citrifolia* seeds is an ingredient in cosmetics.

24. An essential oil according to claim 16, wherein the oil extracted from *Morinda citrifolia* seeds is an ingredient in candles.

25 25. An essential oil according to claim 16, wherein the oil extracted from *Morinda citrifolia* seeds comprises:

0.5 wt. % caprylic fatty acid;

8.4 wt. % palmitic fatty acid;

4.0 wt. % stearic fatty acid;

13.8 wt. % oleic fatty acid;

66.8 wt. % linoleic fatty acid;

0.2 wt. % linolenic fatty acid;

0.5 wt. % arachidic fatty acid; and  
0.2 wt. % eicosenoic fatty acid;

wherein the foregoing concentrations can vary up to  $\pm 10\%$ , by weight.

5 26. A process for extracting oil from *Morinda citrifolia* seeds comprising the steps of:

obtaining *Morinda citrifolia* seeds that are dried to a moisture content less than 10%;  
cracking, flaking, or grinding the seeds to form ground seeds; and  
10 pressing the ground seeds to expel *Morinda citrifolia* oil.

27. A process for extracting oil from *Morinda citrifolia* seeds according to claim 26, further comprising the steps of:

15 mixing the ground seeds with a nonpolar extraction solvent to allow *Morinda citrifolia* oil from the seeds to mix with the extraction solvent; and  
heating the mixture of seeds and extraction solvent to a temperature from 80°F and 200°F.

20 28. A process for extracting oil from *Morinda citrifolia* seeds according to claim 27, wherein the mixture is heated for a period between 30 minutes and 12 hours.

25 29. A process for extracting oil from *Morinda citrifolia* seeds according to claim 27, further comprising the step of removing the extraction solvent from the *Morinda citrifolia* oil by evaporation.

30 30. A process for extracting oil from *Morinda citrifolia* seeds according to claim 27, wherein the extraction solvent is a food-grade extraction solvent.

31. A process for extracting oil from *Morinda citrifolia* seeds according to claim 27, wherein the extraction solvent is hexane.

32. A process for extracting oil from *Morinda citrifolia* seeds according to claim 26, further comprising the step of removing impurities from the *Morinda citrifolia* oil.

33. A process for extracting oil from *Morinda citrifolia* seeds according to claim 32, wherein the impurities are precipitated by addition of a precipitating agent and removed from the *Morinda citrifolia* oil by centrifuge.

5 34. A process for extracting oil from *Morinda citrifolia* seeds according to claim 26, further comprising the step of bleaching the *Morinda citrifolia* oil.

35. A process for extracting oil from *Morinda citrifolia* seeds according to claim 26, further comprising the step of deodorizing the *Morinda citrifolia* oil.

10 36. A process for extracting oil from *Morinda citrifolia* seeds according to claim 26, wherein the seeds are ground to a size between 2 mm to 20 mm.

15 37. A process for extracting oil from *Morinda citrifolia* seeds according to claim 26, further comprising the step of adding an antioxidant to the *Morinda citrifolia* oil.

20 38. A process for extracting oil from *Morinda citrifolia* seeds according to claim 37, wherein the antioxidant is selected from tocopherol acetate (vitamin E acetate), propyl gallate, TBHQ (t-butyl hydroquinone), BHT (3,5-bis(t-butyl)-4-hydroxytoluene), and French maritime pine bark extract.

39. A process for extracting oil from *Morinda citrifolia* seeds comprising the steps of:

25 obtaining *Morinda citrifolia* seeds that are dried to a moisture content less than 10%;

cracking, flaking, or grinding the seeds to form ground seeds having a size in the range from 2 mm to 20 mm;

pressing the ground seeds to expel *Morinda citrifolia* oil;

30 mixing the ground seeds with a food-grade, nonpolar extraction solvent to allow *Morinda citrifolia* oil from the seeds to mix with the extraction solvent;

heating the mixture of seeds and extraction solvent to a temperature from 80°F and 200°F; and

removing impurities from the *Morinda citrifolia* oil.

40. A process for extracting oil from *Morinda citrifolia* seeds according to claim 39, wherein the mixture of seeds and extraction solvent is heated for a period between 30 minutes and 12 hours.

5 41. A process for extracting oil from *Morinda citrifolia* seeds according to claim 39, further comprising the step of removing the extraction solvent from the *Morinda citrifolia* oil by evaporation.

10 42. A process for extracting oil from *Morinda citrifolia* seeds according to claim 39, wherein the extraction solvent is hexane.

15 43. A process for extracting oil from *Morinda citrifolia* seeds according to claim 39, wherein the impurities are precipitated by addition of a precipitating agent and removed from the *Morinda citrifolia* oil by centrifuge.

44. A process for extracting oil from *Morinda citrifolia* seeds according to claim 39, further comprising the step of bleaching the *Morinda citrifolia* oil.

20 45. A process for extracting oil from *Morinda citrifolia* seeds according to claim 39, wherein step of bleaching the *Morinda citrifolia* oil comprises:

adding a filtering agent selected from diatomaceous earth (D.E.) and activated charcoal to the *Morinda citrifolia* oil;

heating the mixture of filtering agent and *Morinda citrifolia* oil to a temperature between 100°F and 200°F; and

25 removing the filter agent by mechanical filtration.

46. A process for extracting oil from *Morinda citrifolia* seeds according to claim 39, further comprising the step of removing volatile impurities by distilling the *Morinda citrifolia* oil.

30 47. A process for extracting oil from *Morinda citrifolia* seeds according to claim 39, further comprising the step of removing residual water by vacuum drying *Morinda citrifolia* oil.

48. A process for extracting oil from *Morinda citrifolia* seeds according to claim 39, further comprising the step of adding an antioxidant to the *Morinda citrifolia* oil.

49. A process for extracting oil from *Morinda citrifolia* seeds according to claim 5 48, wherein the antioxidant is selected from tocopherol acetate (vitamin E acetate), propyl gallate, TBHQ (t-butyl hydroquinone), BHT (3,5-bis(t-butyl)-4-hydroxytoluene), and French maritime pine bark extract.

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US00/23394

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : A01N 65/00

US CL : 424/195.1

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 424/195.1

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,922,766 A (ACOSTA et al) 13 July 1999, see Table I in particular and Examples.	1-14, 16, 17, 22-24
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Y		15, 18-20
X,E	US 6,139,897 A (GOTO et al) 31 October 2000, see Examples in particular for fatty acids, column 5 for disclosure of antioxidant.	1-4, 7-14, 16-17, 20-24
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Y,E		5,6, 15, 18-19
X	US 5,843,499 A (MOREAU et al) 01 December 1998, see column 8 in particular.	1-4, 8, 11-13
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A		25-49

Further documents are listed in the continuation of Box C.  See patent family annex.

* Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
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**INTERNATIONAL SEARCH REPORT**

International application No.  
PCT/US00/23394

**C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,736,174 A (COOPER et al) 07 April 1998, see column 9, lines 52-65.	5-6, 18-19